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N-TYPE MOLYBDENUM DISILENIDE-BASED LIQUID JUNCTION SOLAR CELLS:--ETC(U)
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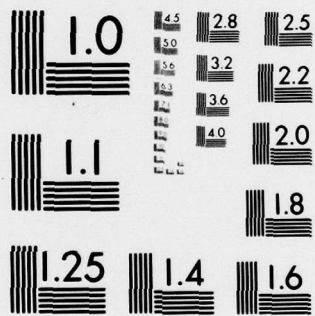
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Single crystal, n-type MoSe₂ ($E_g = 1.4$ eV) is shown to serve as a stable photoanode in an electrochemical cell employing a nonaqueous (CH₃CN) solution of Cl₂/Cl⁻ as the redox active material. At 90 mW/cm² input, 632.8 nm light can be converted to electricity with an efficiency of 5.9-7.5%. The photoanode reaction is 2Cl⁻ → Cl₂ and the cathode reaction is Cl₂ + 2Cl⁻ → 2Cl⁻. The MoSe₂ is qualitatively better than MoS₂ (~0.5% efficiency) which has a larger band gap (1.7 eV), but both materials are rugged in the nonaqueous solution while both photocorrode in aqueous Cl₂ solutions. In H₂O, the I₂/I⁻ couple is excellent

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but in CH_3CN it yields lower efficiency than the Cl_2/Cl^- couple. The stable Cl_2/Cl^- system provides evidence that a transparent, reversible, non- O_2 sensitive redox couple can be useful in n-type semiconductor-based liquid junction cells employing a direct band gap material having optimum solar response.

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6 N-Type Molybdenum Diselenide-Based Liquid Junction Solar Cells:
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by

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N-Type Molybdenum Diselenide-Based Liquid Junction Solar Cells:
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Couple

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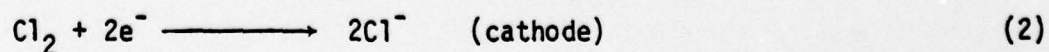
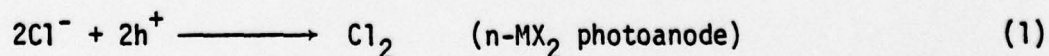
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Abstract: Single crystal, n-type MoSe_2 ($E_g = 1.4 \text{ eV}$) is shown to serve as a stable photoanode in an electrochemical cell employing a nonaqueous (CH_3CN) solution of Cl_2/Cl^- as the redox active material. At 90 mW/cm^2 input, 632.8 nm light can be converted to electricity with an efficiency of $5.9 - 7.5\%$. The photoanode reaction is $2\text{Cl}^- \rightarrow \text{Cl}_2$ and the cathode reaction is $\text{Cl}_2 + 2\text{Cl}^-$. The MoSe_2 is qualitatively better than MoS_2 ($\sim 0.5\%$ efficiency) which has a larger band gap (1.7 eV), but both materials are rugged in the nonaqueous solution while both photocorrode in aqueous Cl^- solutions. In H_2O , the I_3^-/I^- couple is excellent but in CH_3CN it yields lower efficiency than the Cl_2/Cl^- couple. The stable Cl_2/Cl^- system provides evidence that a transparent, reversible, non- O_2 sensitive redox couple can be useful in n-type semiconductor-based liquid junction cells employing a direct band gap material having optimum solar response.

Semiconductor/liquid junction cells for optical energy conversion have been the object of intense study in recent years.¹⁻³ Special emphasis has been placed on the stabilization of small band gap, n-type semiconductors that are generally susceptible to photoanodic corrosion.⁴ The technique of stabilization generally has involved the use of solution redox reagents capable of being oxidized at the n-type semiconducting photoanode material, and considerable progress has been made toward high efficiency. The best efficiency reported to date is ~12% for an n-type GaAs-based cell employing the $\text{Se}_2^{2-}/\text{Se}^{2-}$ redox system in aqueous solution as the stabilizing reagents.⁵ The one generalization that seems to emerge from the stabilization technique is that redox couples giving high efficiency devices are generally at very negative potentials, e.g. $E^\circ(\text{Se}_2^{2-}/\text{Se}^{2-}) \approx -0.9 \text{ V vs. SCE}$.⁶ Such negative potentials mean that the stabilizing reagent is quite unstable with respect to oxidation by O_2 . It is also noteworthy that the stabilizing reagents typically involved have strong visible absorption contributing to inefficiency.

Recently, considerable interest has been directed to MX_2 ($\text{M} = \text{Mo}, \text{W}$; $\text{X} = \text{S}, \text{Se}, \text{Te}$) photoanode materials.⁷ In relation to the potential of the redox couple and its optical absorption, we note that n-type MoS_2 can be used as the photoanode in a non-aqueous electrolyte solution where the electrode reactions are represented by equations (1) and (2).⁸



Surprisingly, the MoS_2 is durable at high photocurrent density ($\sim 10 \text{ mA/cm}^2$) under such harsh conditions; the $E^\circ(\text{Cl}_2/\text{Cl}^-)$ is $+1.1 \text{ V vs. SCE}$ in CH_3CN .⁹ The Cl_2/Cl^- system transmits the entire visible spectrum except for the blue end,

even at high concentration (~ 1 M). The E° for Cl_2/Cl^- is sufficiently positive that Cl^- is not unstable in the presence of O_2 . Unfortunately, the MoS_2 used (natural single crystal) yielded poor overall optical to electrical energy conversion efficiency.

We now wish to report our preliminary findings concerning the Cl_2/Cl^- redox system in conjunction with synthetic, single-crystal, n-type MoSe_2 photoanode-based cells. The MoSe_2 ($E_g = 1.4 \text{ eV}$)¹⁰ has a better band gap than MoS_2 ($E_g = 1.7 \text{ eV}$)¹⁰ for the solar energy conversion application, and we have found several surprising results in using it as a photoanode in non-aqueous electrolyte solutions.

Experimental

Single crystals of MoSe_2 were grown by vapor phase transport using Br_2 as the transport agent. The flat, thin (~ 0.1 mm) crystals were of 0.01 to 0.2 cm^2 in exposed area on each side (001 face). The material was found to be n-type from the photoeffects observed.

Satisfactory electrical contact to the n-type MoSe_2 was made by rubbing Ga-In eutectic onto one side. A copper wire was attached using conducting silver epoxy. The copper wire lead was threaded through 4 mm diameter Pyrex tubing and then the assembly was insulated with ordinary epoxy leaving only the (001) face of the MoSe_2 exposed.

Spectrograde CH_3CN non-aqueous solvent, supporting electrolyte $[\text{Et}_4\text{N}]\text{Cl}$ or $[\text{n-Bu}_4\text{N}]\text{ClO}_4$, and Cl_2 were used as received from commercial sources. All experiments were carried out at 25°C employing single-compartment cells with a working electrode (n- MoSe_2), reference electrode (saturated calomel electrode, SCE), and a counter electrode (generally Pt). The current-voltage data were obtained using a Princeton Applied Research Model 173 potentiostat equipped with a Model 175 programmer; data were recorded with a Houston Instruments Model 2000 x-y recorder.

The light source for the irradiation of n-type MoSe_2 was a He-Ne laser that provided 632.8 nm light of up to ~ 2 W/cm^2 . The power was adjusted by using neutral density glass filters. The incident power on the cell was determined using a Tektronix J 16 digital radiometer equipped with a J6502 probe.

Results

Irradiation (632.8 nm) of n-type MoSe_2 as the photoelectrode in a cell containing the Cl_2/Cl^- couple results in a photocurrent-voltage characteristic as shown in Figure 1. Key output parameters as a function of input optical power for the two representative photoelectrodes are given in Table I. The n-type MoSe_2 , like MoS_2 , is very rugged in the presence of the corrosive Cl_2/Cl^- system. For example, a representative run was done at a photocurrent of 0.8 mA (200 mA/cm^2) for 18.5 h; the current and output parameters remained constant within 5% and no obvious surface damage to MoSe_2 was evident. In such an experiment the entire MoSe_2 crystal was 4 μmol and during the run $\sim 550 \mu\text{mol}$ of electrons passed through the circuit. Thus, high current density can be sustained and it would appear that the chemistry represented by equations (1) and (2) represents the electrode reactions. In the absence of added Cl_2/Cl^- ($[\text{n-Bu}_4\text{N}]\text{ClO}_4$ electrolyte solution only) there is little or no photoanodic current.

The n-type MoSe_2 -based cell is interesting first because such a positive, optically transparent redox couple can be used. Further, the output voltage at open-circuit is quite good, up to 550 mV. The highest output voltage for the MoS_2 was quite similar at similar input optical power.⁸ Thus, the smaller band gap MoSe_2 gives an output voltage about as good as that from MoS_2 . The short-circuit quantum yield, ϕ_e , is in the vicinity of 0.7 for MoSe_2 (no correction for reflection from MoSe_2) whereas the highest value observed for MoS_2 was 0.2 at low light intensity. The fill factor for MoSe_2 is not excellent, but significantly better than the earlier MoS_2 results. The overall efficiency for the conversion of 632.8 nm light to electricity is about an order of magnitude better for the MoSe_2 (5.9-7.5%) compared to MoS_2 ($\sim 0.5\%$).

The differences between MoSe_2 and MoS_2 are not understood. However, we do find that both MoSe_2 and MoS_2 give good behavior in I_3^-/I^- aqueous cells as previously reported.⁷ Thus, the difference between MoSe_2 and MoS_2 in the Cl_2/Cl^- non-aqueous cell is significant and likely intrinsic and not merely a reflection of having poor samples of MoS_2 . That the 1.4 eV response system is better than the 1.7 eV system is quite interesting. The durability of MoSe_2 and MoS_2 appears to be similar. Neither photoanode is durable in aqueous Cl^- solution; presumably the H_2O changes the kinetics and energetics for semiconductor photocorrosion.⁴

Having found that Cl^- oxidation could be sustained by irradiation of the MoS_2 or MoSe_2 we examined the behavior of these photoanodes in the presence of Br^- and I^- in the nonaqueous electrolyte. The E° 's in CH_3CN are +0.70 and +0.20 V vs. SCE for Br_2/Br^- and I_3^-/I^- , respectively.⁹ These are interesting because the I_3^-/I^- system works very well in H_2O . We find that the photoelectrochemical cells based on Br_2/Br^- and I_3^-/I^- in nonaqueous solution are very poor when compared to the Cl_2/Cl^- system. Output voltage, quantum yield, and fill factor are all smaller. In H_2O solvent the I_3^-/I^- system seems to be the best. These results reveal a very important role for solvent in contriving the best liquid junction solar cells.

To summarize our key results, we have found that n-type MoSe_2 serves as a stable photoanode in nonaqueous solutions of Cl_2/Cl^- . Overall efficiency is better than for the larger band gap MoS_2 -based cell, and we find an ordering of efficiency for the I_3^-/I^- , Br_2/Br^- , Cl_2/Cl^- couples that is not found in studies employing aqueous electrolytes. The Cl_2/Cl^- system is transparent and unaffected by O_2 , but it is likely too corrosive to be a practical system. However, the Cl_2/Cl^- cell provides evidence that air stable, reversible, and transparent redox couples can be found for this application.

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Table I. Output Characteristics for an n-Type MoSe₂ Based Photoelectrochemical Cell,^a

	Input, mW/cm ² ^b	ϕ_e^c	Max Power Output, mW/cm ²	Max V (V at η_{max}) ^d	$\eta_{max}, \%^e$	Fill Factor ^f
#1	90.3	0.73	5.31	360 (250)	5.9	0.46
	354	0.65	19.1	410 (250)	5.4	0.41
	839	0.58	44.3	450 (250)	5.3	0.40
	1150	0.55	50.0	450 (250)	4.4	0.34
	2080	0.45	69.0	480 (250)	3.3	0.30
#2	90.3	0.65	6.73	460 (350)	7.5	0.50
	331	0.68	23.4	500 (300)	7.2	0.40
	425	0.66	37.2	540 (250)	4.0	0.22
	1630	0.48	42.5	550 (250)	2.6	0.19

^aData for CH₃CN/1.0 M [Et₄N]Cl with Cl₂ added to bring E_{redox} to +1.05 V vs. SCE for crystal 1 and +0.96 V vs. SCE for crystal 2.

^bInput power is the 632.8 nm line from a Coherent Radiation He-Ne laser.

^cQuantum yield for electron flow at E_{redox}; this corresponds to the short-circuit quantum yield taken as the number of electrons passed per incident photon.

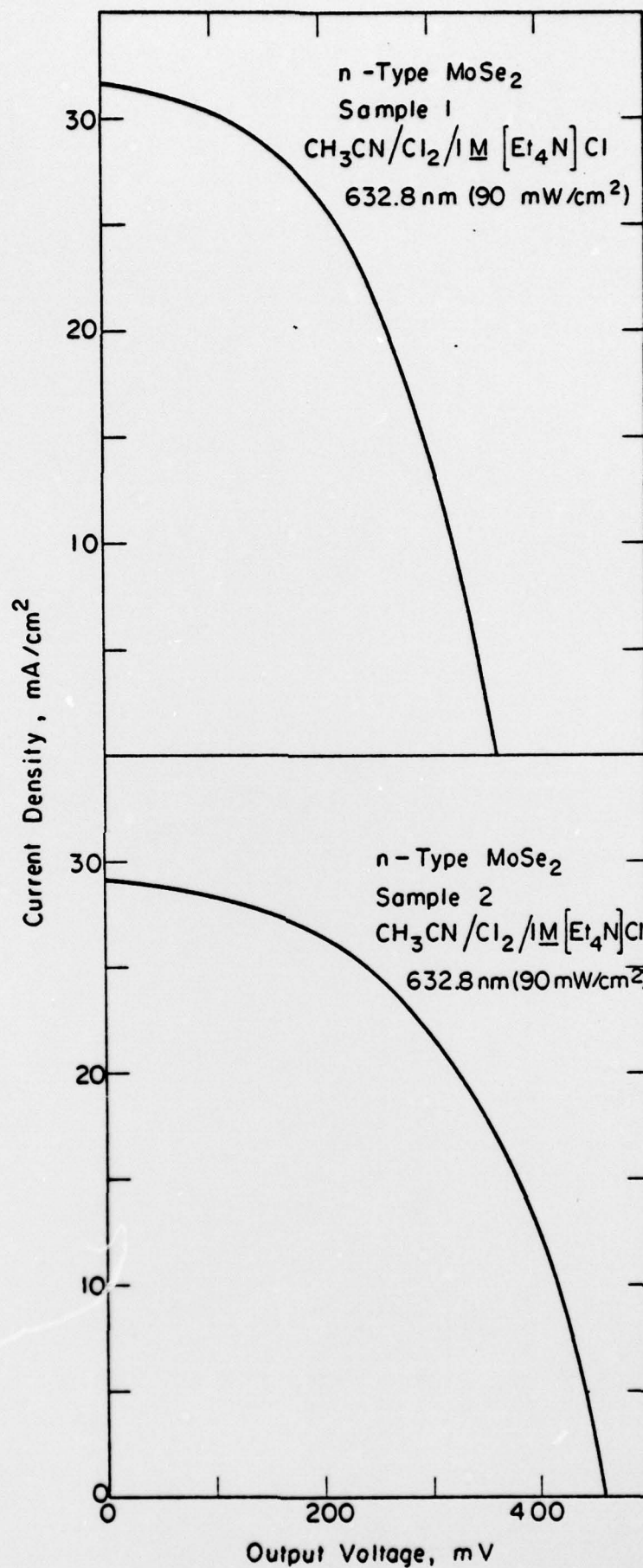
^dMaximum voltage is the open-circuit photopotential and the number in parentheses is the output voltage at the maximum power point.

^eEfficiency for the conversion of 632.8 nm light to electricity.

^fFill factor is a measure of the rectangularity of the current-voltage curves (Figure 1) and is defined as the (max power)/(max V x short-circuit photocurrent).

Figure Caption

Figure 1. Current-voltage curves under 90 mW/cm^2 632.8 nm irradiation for n-MoSe_2 in $\text{CH}_3\text{CN}/1.0 \text{ M } [\text{Et}_4\text{N}]\text{Cl}$ with Cl_2 added to bring E_{redox} to $+1.05 \text{ V}$ vs. SCE for crystal 1 and $+0.96 \text{ V}$ vs. SCE for crystal 2.



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